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DE 42 16 774 A1

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**A process for manufacturing a multi-component granulate**

A process for manufacturing a multi-component granulate for insertion into washing- and cleaning agents, by which in a first stage, a powdered cleaning agent component which is selected from the group of zeolites, alkali phosphates, alkali carbonates, alkali hydrogen carbonates, alkali silicates, laminar silicates or mixtures thereof, is mixed with at least an initial liquid component which contains at least an active cleaning agent ingredient as a binder for the powdered cleaning agent component, and is granulated into granulate particles. In a second stage, the granulate particles which can still be moist, are transferred to a drier, characterised in that the first stage is carried out in a falling stream agglomeration mixer, and in a second stage, the granulate particles emerging from this agglomeration mixer, are immediately transferred to a fluidized bed drier, where they are simultaneously sprayed with a second liquid component which contains at least a cleaning agent ingredient, and are dried there.

**Description**

The submitted invention relates to a process for manufacturing a multi-component granulate.

The overwhelming majority of granular machine washing and rinsing agents found on the market at present, are manufactured from basic materials which are present in granular form. This means that each component is present as a separate granulate. These different granulates are processed into the desired end products in conventional

ways in spray mixing processes, by spraying liquid components through nozzles, such as e.g. surfactants or defoaming agents. Thus the liquid components serve simultaneously for combining the dust and abrasion material.

The classical cleaners contain as granular components, tripolyphosphate, metasilicate, active chlorine distributors such as e.g. dichlorisocyanurate, surfactant and possibly soda, defoamer and fragrances. The pH of a 1% solution of this cleaner in water thereby lies in general between 11 and 12. The performance characteristics are strongly influenced by the particle structure, particle distribution and bulk density of the individual components. In particular during transport or decanting, even already when filling in a production operation, it can give rise to a separation into the individual components, if the named parameters of particle distribution and bulk density are not accurately in agreement with each other. This signifies that the uniformity of the end product can be strongly impaired by subsequent transport or handling, which under unfavourable pre-conditions can lead to strongly varying results in application. This also applies in the same measure to cleaners which come for insertion in commercial washing machines. These cleaners in addition to the already named constituents, also frequently contain sodium hydroxide in the form of fine pearls, in order to increase the alkalinity, because these cleaners frequently have to show a high performance already, frequently with very short retention times of the goods to be cleaned.

In addition to the above named classical cleaners, the so-called low alkaline and compact cleaners have been developed for the machine system and washing. This new generation of cleaning agents can be based not only on phosphate as chelating agent, and also on the combination of organic chelating agents such as citrate, with polycarboxylates as a dispersing agent. Inserted as mild alkali carriers in these products are disilicate and soda, so that the pH of a 1% aqueous solution of this cleaner lies between 10 and 10.5.

Also with these new granular low-alkaline washing- and rinsing agents, the manufacture according to the so-called spray mixture process predominates, and can be carried out in different units of equipment not only continuously but also in batch processes. Examples of such units of equipment named here are mixing types such as ploughshare mixers, double shaft mixers, NAFTA mixers, zig-zag mixers and Telschig mixers. As here also the individual components are present as granulates, the same problems occur as in the case of classical products.

Available for the formulation of classical products in any case for some time, are accurately specified granulates of the main components such as phosphate, metasilicate and soda, so that by a selection of suitable starting materials, the problem of separation during transport can be extensively solved.

The present day raw materials, particularly for phosphate-free cleaners are anyhow not so homogeneous. Thus differences in bulk density of several hundred grams per litre

between a few raw materials can occur. Also the particle structure can vary from round particles, such as e.g. perborate, to broken particles, such as e.g. in the case of soda, up to

angular crystals (e.g. citrate), which leads to the fact that the mixtures are mostly not homogeneous, and strongly tend towards separation.

A process for the manufacture of carbonate-containing granulates is already known from the German Patent application DE-OS-23 22 123. A disadvantage of this process however exists in that only a special, very finely divided sodium carbonate can be inserted.

In order to counter this problem, co-granulates are described in EP-0 488 868, which consist of soda and silicate. These granulates are recovered using a special process, from light soda and waterglass, whereby however the process is limited to the ratio of soda to silicate to a possible range of variations of both starting products, soda and waterglass. The granulates manufactured according to this process can only be prepared to a content of a max. 35% silicate. In addition, the problem also remains of non-uniformity, and a possible separation of the components.

A process for drying granulates is described in DD 228 458. A multi-stage process for the manufacture of a multi-component granulate, is already known from DE-OS-42 16 774. According to this process, several process steps are carried out on a pre-granulate emerging from a ring layer mixer, with subsequently applied fluidized bed drying of the granulate, which is expensive due to a considerable insertion of energy.

Furthermore, EP-A-0403084 discloses a process for manufacturing a zeolite agglomerate, which describes in a first step the agglomeration of zeolite, and in a second step the addition of a detergent, respectively carried out in an agglomeration mixer. This process is also however carried out with a considerable insertion of energy.

A further way of solving this is described in DE-OS-41 01 877, in which a process for manufacturing carbonate-containing granulates is described, whereby the granulation of the components takes place in a mechanism, in which a granulation can be carried out according to one's wishes, with a simultaneous drying of the resulting granulate. However, there is still a disadvantage of the process, in that during filling or transport, the individual granulate particles can break up, and a separation into the individual components can occur.

Moreover in Prior Art there exists the need for granulates with higher bulk densities, which accordingly would facilitate smaller packages with unchanged weight, and thus reduce the expense of packaging. With processes known from Prior Art, a granulate is manufactured conventionally in a spray tower, and possesses a hollow structure and thereby only a lesser density. This granulate is then reduced in size by physical treatment, and then mixed into the final product with further washing- or cleaning agent constituents,

which for example could not necessarily have been processed in a spray tower. This is connected with the already described problem of separation.

The task of the invention thus consists of preparing a process for manufacturing a multi-component granulate for insertion into washing-and cleaning agents, which will yield a granulate in a simple and dependable manner, which excels by an intensified mechanical strength against decomposition, and is not subjected to any separation into the individual components and which will guarantee an outstandingly swift solubility in the washing- or rinsing liquors.

On the part of the Inventor, it was then surprisingly found, that the task of the invention could be solved, by preparing a second stage process, by which in the first stage, a powdered component is granulated in a continuously running agglomeration mixer, with the addition of a liquid component, and in a second stage, the granulate particles which can still be moist are simultaneously sprayed with a second liquid component, and dried in a drying apparatus.

The submitted invention is therefore directed towards a process for manufacturing a multi-component granulate for insertion into washing- and cleaning agents, whereby in a first stage, a powdered cleaning agent component, selected from the group of zeolites, alkali phosphates, alkali carbonates, alkali hydrogen carbonates, alkali silicates, laminar silicates or mixtures thereof, is mixed with at least an initial liquid component which contains an active cleaning agent ingredient which acts as a binder for the powdered cleaning agent component, and is granulated in a continuously running agglomeration mixer into granulate particles. In a second stage, the granulate particles formed, which can still be moist, are transferred to a drying apparatus, whereby the first stage was carried out in a falling stream agglomeration mixer, and in a second stage, the granulate particles emerging from the agglomeration mixer are immediately transferred to a fluidized bed drier and can undergo simultaneous spraying with a second liquid component which contains at least a cleaning agent ingredient, and is followed by drying.

Understood under cleaning agent components in the embodiment of the invention, is every type of substance which can find application in conventional washing- and/or cleaning agents. Under the expression moist in the embodiment of the invention, is understood to be that the granulate particles can have a content of liquid component(s) used in the first stage. In the process in the embodiment of the invention, a powdered component is agglomerated into a moist granulate in an agglomerating mixer of the continuous type, with the addition of a liquid component. The still moist granulate is transferred to a drier with simultaneous spraying with a second liquid component, then dried with hot air, preferably in such a way that owing to the heat of evaporation of the granulate during the drying it is held at a temperature below the temperature of the hot air fed in ("adiabatic drying").

The powdered component which as a rule is the substance used as matrix substance for the liquid component, or can be used as a cleaning agent constituent dissolved in it, and itself can positively influence the cleaning process, having alkali-, surfactant- or chelating properties, can be selected for example from the group consisting

of zeolites, alkali phosphates, particularly penta-sodium triphosphate, alkali carbonates, alkali hydrogen carbonates, alkali silicates, laminar silicates or mixtures of them.

The powdered components, whose particle size is not decisive, as along as an adjustment can be made to the desired particle size and bulk density of the finished multi-component granulates, are preferably dispensed as a rule from those above, using dispensing devices, into the agglomeration mixer. The descending powder particles are sprayed for example from the side, with at least one liquid component, and agglomerated into granulate particles. Two different liquid components can also be used here, and obviously can also be used individually, whereby one can be an aqueous solution of constituents conventionally contained in cleaning agents, such as dispersing agents, surfactants, chelating agents or alkali silicates, and the other can be an anhydrous solution of surfactants such as e.g. linear alkyl benzene sulphonates. The quantity of liquid component(s) which can be mixed into the first stage, in dependence of the quantity of powdered cleaning agent components used, can be selected to be so large, until a further addition gives rise to a clumping of the powdered component, and a type of "pulpy formation" can be observed in the agglomerating mixer.

The powdered components and the quantity of liquid components can be accurately dispensed in the first stage over a dispensing system. Through a suitable selection of the process parameter for the corresponding insertion, the particle structure, particle distribution and bulk density of the granulate manufactured according to the process in the embodiment of the invention, can be adjusted over a wide range. The quantity of liquid component which is added in the first stage, and its viscosity, can influence the construction of the granulate over a wide range. Thus in the first stage of the process, 10 to 90, preferably 20 to 80% by weight of the total weight of inserted liquid components are used.

A falling stream agglomeration mixer is used as an agglomeration mixer, which is in the position of being able to construct a granulate. A self-cleaning down-pipe mixer is preferably inserted, with an internal rotor, as this does not tend to become clogged up, and facilitates a large throughput with comparatively small dimensions. The mixing and agglomeration of the components are thus achieved above all by centrifugal force, and the mechanics of the quickly rotating knife.

The granulate particles formed in the agglomeration mixer and emerge preferably from this exposure to the force of gravity, are transferred to the drying apparatus. In the simplest case, the agglomeration mixer is located above the drying apparatus, and the granulate particles fall directly into the drying apparatus. A fluidized bed drier is preferably

used as drying apparatus, so that the granulate particles fall directly out of the agglomeration mixer, into the fluidized bed, and are held in suspension by the air blown in from below, and become dried. It is thus possible to prevent caking of the granulate particles, which can still be moist, and forming a non-homogeneous or coarse particle granulate.

The process in the embodiment of the invention can be carried out in a wide temperature range, limited at the top by the decomposition temperature of one of the components used, e.g. the bleaching agent, and at the bottom by the ease of flow of the liquid component.

Simultaneously in the drying apparatus in this second stage, with simultaneous drying in hot air, a second liquid component is sprayed through nozzles on to the granulate particles, which can still be moist. The quantity of liquid component sprayed on through nozzles can be selected to be so large, in dependence of the quantity of granulate particles, that the granulate particles can still be held in suspension, without forming clumps. For this purpose it can be necessary if required, to increase the quantity of air fed in from below for drying the granulate particles.

The second liquid component can thus have a different or the same composition as the first liquid component. It can also consist for example of an aqueous solution of a solid, which is already present in the granulate in solid form. Thus for example for manufacturing a granulate for insertion into dishwashing, sodium carbonate in the form of calcined soda is granulated with silicate present in the form of waterglass, so that a desired particle results, and then in the second stage, so much waterglass can be added by spraying, with simultaneous drying, so that the desired ratio of silicate to soda is obtained. The degree of drying can be thus adapted in such a way that the speed of dissolving of the granulate in the wash liquor is not adversely influenced.

Thus by dissolving a second liquid component, the applied quantity of solid on the powdered component is further raised, and correspondingly a bulk density is achieved, which cannot be reached by known processes in Prior Art. At present in Prior Art, a maximum bulk density of 700 - 750 g/l is reached, whilst with the process in the embodiment of the invention, those of more than 800 g/l, preferably even more than 850 g/l can be achieved. Simultaneously the speed of dissolving of the granulate in the wash liquor is not adversely influenced, so that when mixing in further components in the final production step, which is connected to the drying stage, in which components particularly sensitive to temperature or the presence of other ingredients, such as bleaching agent activators, enzymes or fragrances can be mixed in, so as to obtain completed cleaning agents with high bulk density, the so-called compact cleaners, which permit smaller packaging units to be used. Simultaneously when manufacturing multi-component granulates, a large saving of energy can be achieved, by dispensing with drying towers.

The nucleus of the end products manufactured according to the process in the embodiment of the invention, consists of a mixture of starting products intimately mixed in an agglomeration mixer, whilst the shell surrounding the nucleus has a layered construction of constituents dissolved or dispersed in the liquid component. In the above named example of soda/silicate, the nucleus consists of a mixture of soda and silicate, whilst the layer almost completely surrounding the nucleus, consists of silicate. Thus for example it is also possible to mix substances as for example fragrances or enzymes, which

can be sensitive to oxidation- or light, and which can decompose during storage of the completed granulate, into the first stage in the mixing of the nucleus, and then to protect them from such destruction processes by the encapsulating layer introduced in the second stage.

According to each product desired there unfolds for the Expert a series of possibilities as regards which components can be inserted in which stage of the process. A few components present themselves as powdered components, which can be granulated after mixing in the liquid components. It is likewise conceivable that for manufacturing a granulate as a powder, components submitted for manufacturing another granulate in dissolved form, for example as an aqueous solution, are sprayed on a powdered component. The exact sequence of being present or addition of the components are selected by the Expert with a view to the designation of the granulate. The components inserted for this purpose are known conventionally in Prior Art for use in Cleaning agents, and their corresponding selection is encountered by the Expert according to known criteria.

Conventional ingredients known from Prior Art for washing-, rinsing- and cleaning agents can be used as further constituents of the multi-component granulates. For this purpose there belong surfactants, bleaching agents, foam inhibitors, carboxymethyl cellulose, methylcellulose, polyvinylpyrrolidone, dyestuffs and dispersing agents.

The implementations of the sub-Claims relate preferably to the implementations of the process for manufacturing multi-component granulates for specified insertion purposes.

Processes for manufacture of granulates are thus preferred, which find application as water-softening additives in washing- and cleaning agents. Preferably used for this purpose as powdered components of a cleaning agent component with builder properties, are those selected from the group consisting of zeolites, alkali carbonates, alkali silicates, laminar silicates, or mixtures thereof. These builder components which should also possess in addition the properties supporting washing activity, are mixed and granulated in an agglomeration mixer, using an aqueous solution simultaneously as a binder for the powdered active substance component, selected from the group consisting of polymeric dispersing agents, organic chelating agents, or mixtures thereof. For simplification of the process, identical solutions are preferably used as the first and second liquid components.

Particularly preferred to be used for this purpose as dispersing agents, are polymerisates and co-polymerisates of acrylic acid, methacrylic acid and maleic acid and their esters, as well as mixtures thereof, and also polyamino acids, and as organic chelating agents, nitrilo triacetic acid, citrates, alkali phosphonates, or mixtures thereof. By the selection of solid components and the constituents of co-granulation, and the second liquid component, multi-component granulates can be manufactured for use as softening additives, which are composed for example from 40 - 95% by weight inorganic ion exchangers such as zeolites, which optionally can contain up to about 20% by weight water of crystallisation such as is the case with zeolite A, or laminar silicates or mixtures thereof, plus 2 - 30% by

weight of the named polymeric dispersing agent, organic chelating agent or mixtures of these, as well as up to 50 % by weight soda, sodium sesquicarbonate or alkali phosphonates.

Further preferred implementations of the process in the embodiment of the invention are directed towards the manufacture of multi-component granulates for granular machine dishwashing agents. Not only phosphate-free but also phosphate containing granulates are manufactured for this purpose. Thus the implementation according to Claim 6 is directed towards a process by which in the first stage, a powdered cleaning agent component which contains alkali carbonate, alkali hydrogen carbonate, alkali phosphate or mixtures of these, is granulated by mixing in an agglomeration mixer at least an initial liquid component consisting of an aqueous solution of alkali silicate with a molar ratio of  $\text{SiO}_2 : \text{Me}_2\text{O}$  of more than 1.0 : 1, preferably more than 1.5 : 1 whereby Me stands for an alkali metal, and then in a second stage, the granulate particles, which can still be moist, are transferred to a drying apparatus, and then subjected to simultaneous spraying in the drying apparatus with a second liquid component which contains at least one cleaning agent ingredient, and then dried. The second liquid component preferably possesses the same composition as the first liquid component.

Used as alkali carbonate or alkali hydrogen carbonate in the embodiment of the invention, are preferably soda, sodium hydrogen carbonate and -sesquicarbonate in powder form, especially with a bulk density of 500 to 1000 g/l.

As a rule, the first liquid component here has a content of alkali silicate of 20 to 55, preferably 40 to 45% by weight. Preferably used as an alkali silicate in the embodiment of the invention, is sodium silicate with a molar ratio of  $\text{SiO}_2 : \text{Me}_2\text{O}$  of more than 1.0 : 1, preferably more than 1.5 : 1, and particularly preferred greater than 1.8 : 1.

The additional ingredients optionally contained in the finished granulate, can be present according to each suitability, type or availability, as powdered components or as one of the liquid components.

In a simple implementation of the process of the invention, calcined soda in the form of powder is granulated in a granulation mixer with high centrifugal force, with a

waterglass of average viscosity of a few hundred to a few thousand m.Pas (35 -45% by weight disilicate). The still moist granulate falls directly into a multi-stage fluidized bed drier. In the first chamber of the fluidized bed drier, further water glass of the same type is sprayed in, with simultaneous drying with hot air which is blown in through the sieve diaphragm from below.

Prepared with this process are preferably multi-component granulates with the following contents according to process Claim 7. A phosphate-free multi-component granulate manufactured according to the process in the embodiment of the invention, thus has a preferable content according to process Claim 8. A further phosphate-containing

multi-component granulate manufactured according to the process in the embodiment of the invention, thus has a preferable content according to process Claim 9.

According to another preferred implementation of the process in the embodiment of the invention, multi-component granulates can thereby be manufactured, particularly for insertion into detergent powders with high bulk density and low dosage requirements, by using in a first stage a powdered component containing alkali carbonate, alkali hydrogen carbonate, alkali silicate or mixtures thereof, and a further powdered ingredient selected from zeolites, laminar silicates, alkali phosphates, or a mixture of zeolites and laminar silicates, and optionally a bleaching agent component, and mixing in an initial liquid component selected from an anhydrous surfactant composition and/or an aqueous solution of polymeric dispersing agents, and granulating in a continuously running agglomeration mixer. In a second stage, the granulate particles which can still be moist, are transferred to a drying apparatus, and simultaneously sprayed there with a second liquid component of an aqueous solution of polymeric dispersing agents, and dried there.

The multi-component granulates manufactured according to the process in the embodiment of the invention for preferred insertion into detergent compact cleaners, have the following contents:

10 -50% by weight builder components such as zeolites, phosphates, laminar silicates or mixtures thereof,  
5 -30% by weight alkali carbonate, -hydrogen carbonate or mixtures thereof, particularly soda:  
5 -30% by weight bleaching agent, preferably perborate as monohydrate;  
3 -20% by weight non-ionic surfactant, particularly fatty alcohol ethoxylate;  
3 -20% by weight anionic surfactant, particularly linear alkyl benzene sulphonate;  
2 -15% by weight dispersing agent, particularly polycarboxylates and other components such as phosphonates, which are conventionally contained in detergents;  
2 -15% by weight water as water of crystallisation or water combined in structural layers.

Used as surfactant components in the embodiment of the invention, are non-ionic, anionic or cationic surfactants. The use is preferred of an anhydrous solution of linear alkyl benzene sulphonates with acid properties, where as a consequence of neutralisation

between the acid groups of the surfactants and the powdered alkali components, the resulting heat of reaction can be used, in order to reduce the quantity of heat needed to generate the hot air fed in for drying in the second stage. In all cases, the process in the embodiment of the invention gives rise to products with very good properties such as rinsing-in ability, dissolving capability and free-flowing ability.

The submitted invention is thus also preferably directed towards a multi-component granulate manufactured according to the process in the embodiment of the invention, whereby an individual multi-component granulate particle is characterised by it having a nucleus which is almost completely encapsulated by a coating, whereby the nucleus essentially consists of a co-granulate of a powdered component containing zeolites, alkali phosphates, alkali carbonates, alkali hydrogen carbonates, alkali silicates or

mixtures thereof, with a binding agent component for the powdered component selected from the group which consists of polymeric dispersing agents, water-soluble alkali silicates with a molar ratio of  $\text{SiO}_2 : \text{Me}_2\text{O}$  of greater than 1.0 : 1, preferably more than 1.5 : 1, whereby Me stands for an alkali metal, or mixtures of these.

The coating encapsulating the nucleus is essentially selected as a substance selected from the group of polymeric dispersing agents, water-soluble alkali silicates with a molar ratio of  $\text{SiO}_2 : \text{Me}_2\text{O}$  greater than 1.0 : 1, preferably more than 1.5 : 1, whereby Me stands for an alkali metal or mixtures of these.

The nucleus and/or coating, contain optional further constituents, selected from dispersing agents, chelating agents, selected from the groups of phosphates or organic chelating agents, auxiliaries selected from the group of bleaching agents, foam inhibitors, methyl cellulose, carboxymethylcellulose, polyvinylpyrrolidone and dyestuffs and water.

The multi-component granulate can also be designed in such a way, that it has a nucleus and at least an almost complete coating encapsulating the nucleus, whereby the nucleus consists of a co-granulate formed from a powdered component which contains calcined alkali carbonate, alkali hydrogen carbonate or mixtures thereof, and a liquid component which contains the alkali silicate with a molar ratio of  $\text{SiO}_2 : \text{Me}_2\text{O}$  greater than 1.0 : 1, preferably more than 1.5 : 1, whereby Me stands for an alkali metal, and whereby the nucleus and/or the coating optionally contains further constituents selected from dispersing agents, chelating agents selected from the group of phosphates or organic chelating agents, auxiliaries selected from the group of bleaching agents, foam inhibitors, carboxymethylcellulose, methylcellulose, polyvinylpyrrolidone and dyestuffs, and water.

#### Manufacture- and comparative examples (subsequently supplied)

By the following manufacture of all examples of powder components, which can also contain a portion of particulate material, it involves metering over proportioning belt weighers into the swiftly rotating continuously running agglomeration mixer. The liquids or liquid mixtures needed are added correspondingly to the mixer over separate metering

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systems. In the mixer, they are very finely distributed through binary nozzles with air, and then mixed with the powdered components by the mechanical influence of the mixer. The mixing tools, such as the knife of the mixer, are introduced in such a way, so as to generate an intimate turbulence and compacting of the quantity under rotation, and agglomeration of the individual components. The retention time in the mixer thus amounts to less than 1 second. The mixer is thus positioned over a continuously operating fluidized bed drier, so that the moist agglomerate falls directly into the fluidized bed, and can be dried there with a stream of air. If needed, the air stream can be heated.

##### 1) Combination granulate silicate/soda

Inserted in both examples as soda, is a light anhydrous powder with a bulk density of about 500 g/l. The waterglass 50/52 was an aqueous solution of 45% by weight sodium silicate ( $\text{Na}_2\text{O} : \text{SiO}_2 = 2.0 \pm 0.1$ ).

### a) Comparative Example

In a swiftly rotating, continuously running agglomeration mixer, the following raw materials were agglomerated according to the above data:

Material: (added to the mixer)	output per hour
Soda (sodium carbonate)	5650 kg
Waterglass 50/52	4000 kg
Rotation speed of mixer	2500 RPM

The temperature of the waterglass during metering was about 40°C. The agglomerate fell directly into the fluidized bed, and was dried there with an inlet air temperature of 180°C, to a final moisture content of 10% water. The retention time in the fluidized bed, inclusive of a cooling phase at the end of drying, amounted to about 30 minutes. The product was free-flowing and did not clump. The bulk density of the agent amounted to 700 g/l. The content of silicate, anhydrous, amounted to 22%. The product is not completely soluble in distilled water, presumably because of a partial decomposition of the silicate to insoluble silicic acid.

### b) Manufacturing Example

The following raw materials were agglomerated in a swiftly rotating, continuously running agglomeration mixer, according to the above data :

Material: (added to the mixer)	output per hour
Soda (sodium carbonate)	2700 kg
Waterglass 50/52 (45% by weight)	1710 kg
Rotation speed of mixer:	2800 RPM

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The temperature of the waterglass during metering lay at 40°C. The agglomerate fell directly into the fluidized bed, and was dried there at an input air temperature of 80°C. By sufficient filling of the fluidized bed, simultaneously during the drying in the fluidized bed, 1755 kg/h waterglass was sprayed in through binary nozzles on to the agglomerate. The retention time in the fluidized bed inclusive of a cooling phase at the end of the drying was about 2 hours. The product was free-flowing and did not clump; the final moisture amounted to 10%. The bulk density of the agent amounted to 870 g/l. The content of silicate anhydrous amounted to 33%. The granulate is quickly completely soluble. Without the two-stage process, one can achieve neither the high bulk density, nor the high content of silicate.

### 2) Manufacture of a detergent powder compound with higher bulk density

The following raw materials were agglomerated in a swiftly rotating agglomerating mixer, according to the above data:

Material: (added in the mixer)	output per hour
Soda (sodium carbonate)	1000 kg
Zeolite A powder	1700 kg
Perborate monohydrate	1100 kg
Fatty alcohol ethoxylate	600 kg
Linear dodecyl benzene sulphonic acid	500 kg
Co-polymer (Sokalan CP 5® of BASF)	300 kg
Water	200 kg

The temperature of the liquids was adjusted to 40°C, before they were sprayed through nozzles into the mixer. The agglomerate fell directly into the fluidized bed, and dried there at an air inlet temperature of 80°C. During the agglomeration in the mixer and the drying in the fluidized bed, the dodecyl benzene sulphonic acid reacted with the soda, with the formation of bicarbonate and dodecyl benzene sulphate. By this mode of procedure, a granulate resulted, which was subjected to the action of dry air in the fluidized bed, and was subsequently compacted to a high bulk density by the turbulence thereby involved. At the end of the drying part of the fluidized bed, shortly before the cooling zone, the granulate which still had an output of 300 kg/h, was sprayed with a solution of the named co-polymer diluted to a content of 25%. The total retention time of the granulate in the fluidized bed, amounted to about 1 hour.

By this manufacture in the embodiment of the invention, solid granulates were formed, which in spite of a high content of fatty alcohol ethoxylate, was not sticky. The flowing process of the granulate was extraordinarily good. The rinsing-in behaviour in the metering chamber of the washing machine, and speed of dissolving in the washing machine were good to very good. The bulk density of the compound was at least 870 g/l. The

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product hardly contained any fine particles and is coarsely grained with an average particle size of 2.5 mm.

#### Patent Claims

1. A process for manufacturing a multi-component granulate for insertion into washing- and cleaning agents, by which in a first stage, a powdered cleaning agent component which is selected from the group of zeolites, alkali phosphates, alkali carbonates, alkali hydrogen carbonates, alkali silicates, laminar silicates or mixtures thereof, is mixed with at least an initial liquid component which contains at least an active cleaning agent ingredient as a binder for the powdered cleaning agent component, and is granulated into granulate particles. In a second stage, the granulate particles which can still be moist, are transferred to a drier, characterised in that the first stage is carried out in a falling stream agglomeration mixer, and in a second stage, the granulate particles emerging from this agglomeration mixer, are immediately transferred to a fluidized bed drier, where they are simultaneously

sprayed with a second liquid component which contains at least a cleaning agent ingredient, and are dried there.

2. A process as Claim 1, characterised in that the granulate particles emerging from the agglomeration mixer, are immediately transferred to a fluidized bed drier, by the action of gravity.
3. A process as Claims 1 or 2, characterised in that the first liquid component has the same composition as that of the second liquid component.
4. A process as Claim 3, characterised in that the first and/or second liquid component is an aqueous solution of an active substance used as a binder for the powdered component, and is selected from the group which consists of polymeric dispersing agents, organic chelating agents or mixtures thereof.
5. A process as Claim 4, characterised in that used as polymeric dispersing agents, are polymerisates and co-polymerisates of acrylic acid, methacrylic acid and maleic acid and their esters, as well as mixtures thereof, and also polyamino acids; and as organic chelating agents, nitrilotriacetic acid, citrates, alkali phosphonates or mixtures thereof.
6. A process as one of Claims 1-5, characterised in that the powdered cleaning agent component contains alkali carbonate, alkali hydrogen carbonate, alkali phosphate or mixtures thereof, and the initial liquid component consists of an aqueous solution of alkali silicate with a molar ratio of  $\text{SiO}_2 : \text{Me}_2\text{O}$  larger than 1.0 : 1, preferably more than 1.5 : 1, in which Me stands for an alkali metal.

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7. A process as Claim 6 for manufacturing a multi-component granulate for insertion into washing- and cleaning agents, with the following contents:
  - 10 -90% by weight alkali carbonate, -hydrogen carbonate or mixtures thereof,
  - 5 - 50% by weight alkali silicate with a molar ration of  $\text{SiO}_2 : \text{Me}_2\text{O}$  greater than 1.0 : 1, preferably 1.5 : 1;
  - 0 -20% by weight polymeric dispersing agent,
  - 0- 50% by weight chelating agent, selected from the group of phosphates or organic chelating agents;
  - 0 - 20% by weight auxiliaries selected from the group of bleaching agents, foam inhibitors, carboxymethylcellulose, polyvinylpyrrolidone and dyestuffs; and
  - 3 -15% by weight water,

whereby the total parts by weight of the individual components in the multi-component granulate, amounts to 100% by weight.
8. A process as Claim 7 for manufacturing a multi-component granulate for insertion into washing- and cleaning agents, with the following contents: